

H. Matino
T. Ushiroda

Effect of Substrate Bias on Properties of RF-Sputtered Cr-SiO Films

Abstract: Properties of rf-sputtered Cr-SiO cermet films have been studied as a function of rf substrate bias. Films with five orders-of-magnitude change in electrical resistivity have been deposited from one Cr-SiO(50:50 wt %) target by changing the substrate bias. Resistivities of about $200 \Omega \cdot \text{cm}$ at 5% bias and about $2 \times 10^{-3} \Omega \cdot \text{cm}$ at 20% bias have been obtained.

Introduction

Cr-SiO cermets, because of their high specific resistivity and excellent stability even for high sheet resistance [1], are important in electronic device fabrication as resistor materials in discrete or integrated components. Several methods have been commonly used for the deposition of Cr-SiO cermet layers, including co-sputtering by dual targets [2], co-evaporation from dual sources [3], and flash evaporation [4, 5]. Unfortunately, these methods require the use of various content sources or targets in order to achieve a wide range of resistances, since the resistivity of films increases with decreasing metal content. The resistivities obtained by flash evaporation or by co-evaporation are different even when using the same content sources. Resistivities of films deposited by flash evaporation are lower than those of films deposited by co-evaporation or co-sputtering except in the case of small SiO content [2, 3, 5]. In this investigation, Cr-SiO films are sputtered at 13.56 MHz with rf substrate bias. The effect of the bias on the properties of sputtered Cr-SiO films and the control of sheet resistance by the bias level are described.

Experimental

The Cr-SiO films were deposited directly on substrates of Corning 7059 glass, glazed alumina, or polished alumina plates that nearly cover the substrate holder, by using a 20.3-cm-diameter hot-pressed Cr-SiO (50:50 wt %) target obtained from Materials Research Corporation, Orangeburg, NY. The depositions were carried out in a conventional planar rf sputtering system. The distance between the anode and cathode was 5.1 cm. The substrates were biased by inserting a tuning network

between the substrate holder and ground [6]. Initially, the chamber was pumped down to at least 5×10^{-7} torr (6.67×10^{-5} Pa) in order to eliminate the effects of residual gas contamination, and then the pumping system and an argon inlet valve were throttled to maintain the admitted sputtering gas pressure at approximately 5×10^{-3} torr (6.67×10^{-1} Pa). The principal variable in this investigation was the substrate bias percentage from 0 to 20% of the target voltage, which is 0 to 325 V (peak to peak) at 1.0 kW and 0 to 230 V at 0.5 kW rf power.

A photolithographic technique was used in measuring film thicknesses. On those samples used for the thickness evaluations, a gold layer 1000 Å thick was deposited under the Cr-SiO films in order to eliminate etching of the substrate by the Cr-SiO etchant. A stripe pattern of photoresist was formed on the Cr-SiO films. The unprotected Cr-SiO was removed using an etchant consisting of 20 ml buffered hydrofluoric acid solution [7], 30 ml of concentrated nitric acid, and 70 ml of deionized water. A 1000-Å aluminum film was then evaporated onto the sample surface, and the thickness of the Cr-SiO films measured by interferometry. On the samples used for electrical measurements, a gold layer 3000 Å thick, to be used as an electrode, was deposited on the Cr-SiO film and was heated at 500°C (773 K) for 20 min in a vacuum of 10^{-1} Pa. The electrical resistance of the samples was measured digitally by means of a four-point probe. Ohmic contact between the Cr-SiO and the gold electrode was maintained even in highly resistive films, as evidenced by the fact that the current-voltage characteristics, as observed by a curve tracer, remained completely linear over several orders of magnitude of current.

Results

• Electrical properties

Figure 1 shows the specific resistivity measured at room temperature as a function of substrate bias voltage. It can be seen that the resistivity depends strongly upon the percent bias applied rather than upon the actual voltage. The resistivity measurements were made on films more than $0.4 \mu\text{m}$ thick in order to eliminate the gettering effect [8]. In films over $3000 \text{ \AA}$ thick, measured specific resistivities were constant under the same bias conditions. The resistivity decreases from about $200 \Omega \cdot \text{cm}$ for a substrate bias of 5% to about $2 \times 10^{-3} \Omega \cdot \text{cm}$ for a bias of 20%. Variation of resistance as a function of $1000/T(\text{K}^{-1})$ for 5, 10, 12.5, 17.5, and 20% bias-sputtered films is shown in Fig. 2. The temperature coefficient of resistance is -2000 ppm even on $200 \Omega \cdot \text{cm}$ films (equivalent to $20 \text{ M}\Omega \cdot \text{cm}^{-2}$ sheet resistance for a film $1000 \text{ \AA}$ thick). These high sheet resistances and low temperature coefficient of resistance values are not obtained in the implanted or diffused resistors on silicon [9].

• Heat treatment

Figures 3(a) and 3(b) show the resistance change caused by heat treatment for 20 minutes in air and in a vacuum of 10^{-1} Pa as a function of the deposition conditions. Significant resistance changes appeared when the temperature exceeded 400°C (673 K). Relatively larger changes were observed for heat treatment in air as opposed to under vacuum. The 12.5 to 15% bias films in both cases show only small changes in resistance. Decreasing resistance was observed in the over 15% bias films, and suggests that the formation of Cr_3Si , rather than oxidation [10], is the dominant factor involved.

• Etching properties

Figure 4 illustrates the variation in etch rate with substrate bias. The etch rate decreases by about two orders of magnitude as the bias is increased from 0 to 20%, and gives a maximum value for a 2.5% bias, similar to the resistivity vs bias data. Freshly made etchant, consisting of 20 ml buffered hydrofluoric acid solution [7], 30 ml concentrated nitric acid, and 70 ml deionized water, was used for these experiments.

• X-Ray microanalysis

X-Ray microanalysis measurements were made on films deposited on glass substrates at 5% and 17.5% bias levels. The 17.5% bias films exhibited a 15 to 25% increase in CrK_β intensities and a 10 to 30% reduction in Si and oxygen K_α intensities in comparison with the 5% films. Thus, the Cr content of films deposited with 17.5% bias was larger than that of the 5% bias films, while the Si and oxygen contents of the 17.5% bias films were less

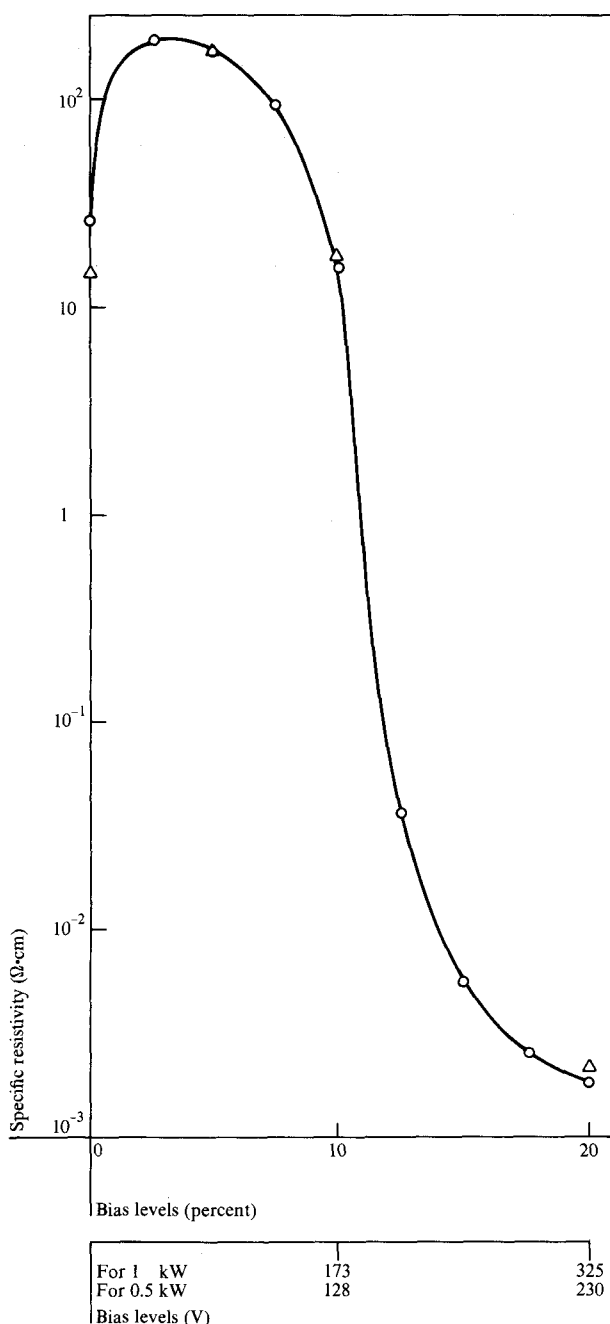


Figure 1 Room temperature specific resistivity of Cr-SiO cermet films shown as a function of substrate bias voltage (peak to peak) and of the percent bias level for 0.5 kW (Δ) and 1.0 kW ($\circ$) rf power levels.

than those of the 5% bias films. Similar results were obtained for films deposited on alumina substrates.

Application

For thermal printing heads, thin-film NiCr and Ta_2N have been widely used as heating element materials. The specific resistivities of NiCr and Ta_2N films are about

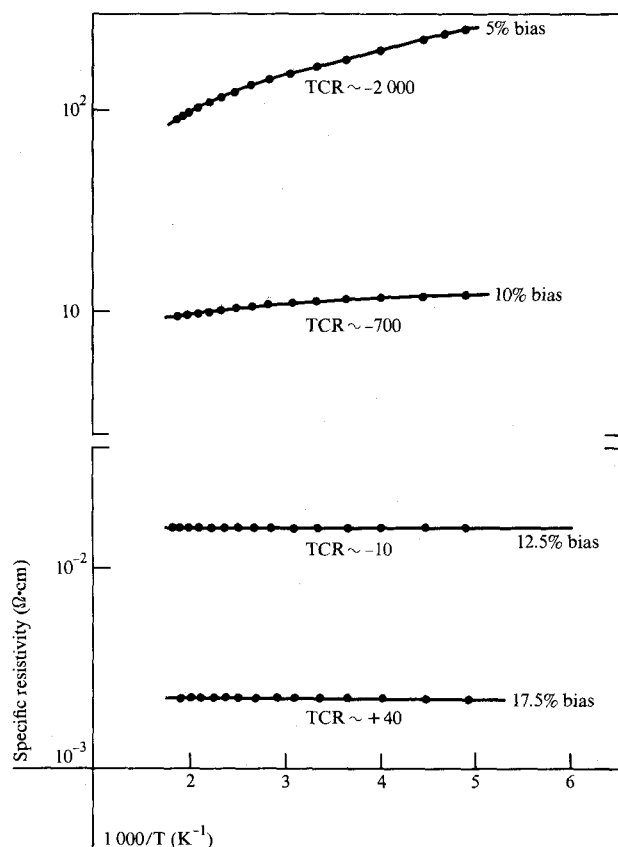


Figure 2 Variation of the specific resistivity with substrate temperature for Cr-SiO films prepared at different bias voltages at 1.0 kW. Values shown for the temperature coefficient of resistance (TCR) are given in ppm/K.

1.5×10^{-4} and $2.4 \times 10^{-4} \Omega \cdot \text{cm}$, respectively, while for a $50 \Omega \cdot \text{cm}^{-2}$ sheet resistance, the required thicknesses of these films are 300 Å and 480 Å, respectively. The current density through these films reaches about $1 \times 10^6 \text{ A} \cdot \text{cm}^{-2}$ under actual operating conditions, and this value is sufficiently high to cause electromigration effects. The thermal print heads using Cr-SiO films, however, have improved reliability because of reduced electromigration effects. This is principally due to the reduced current density, since for the same sheet resistance, the Cr-SiO films are thicker than NiCr or Ta₂N films. In preliminary experiments Cr-SiO heads, which were covered with a SiO₂ overcoating layer 2 μm thick, showed a factor-of-three improvement over Ta₂N heads in the mean times to failure (failure being defined as a resistance change greater than ±10%).

Conclusions

The results presented here indicate that the properties of rf-sputtered Cr-SiO cermet films depend strongly upon the substrate bias used during deposition. The use of con-

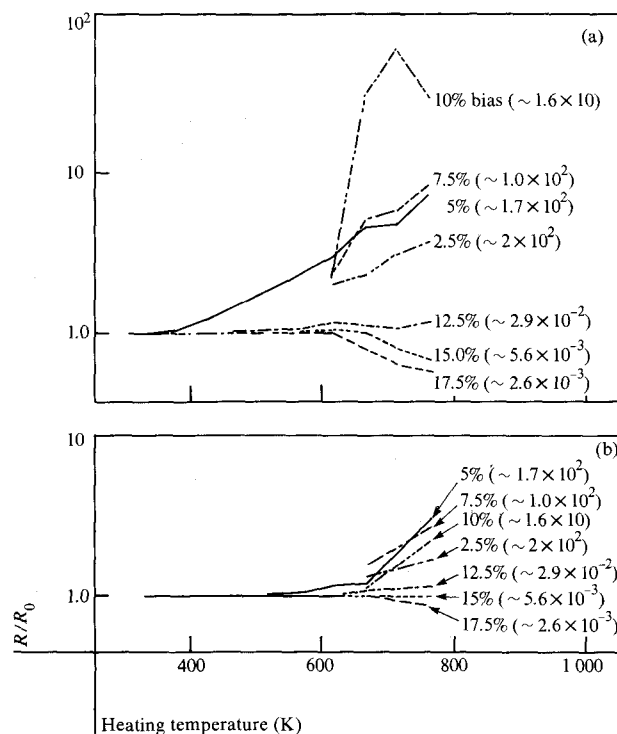


Figure 3 The effect of bias voltage on the change in film resistance with heat treatment (a) in air and (b) under a vacuum of 10^{-1} Pa. Vertical axis is plotted as the ratio of final heat-treated resistance R to the initial room temperature resistance, R_0 . Parenthetical values refer to $R_0 \Omega \cdot \text{cm}$ for films prepared at different bias levels.

trolled substrate bias has enabled us to obtain, from a single Cr-SiO target (50:50 wt %), Cr-SiO films with sheet resistances that can be varied over five orders of magnitude. The results show that the resistivity of deposited films decreases as the substrate bias is increased. This is attributed to changes in the Cr/Si and Cr/O ratios in the sputtered films, which are in turn determined by the substrate bias ratio.

Acknowledgment

The authors thank N. Amemiya of the IBM Japan Yasu plant, for his contributions to the x-ray microanalyses.

References and note

1. M. Beckerman and R. L. Bullard, "Integrally Fabricated Resistors and Their Performance," *Proceedings of the Electronic Components Conference*, Washington, DC, May 8-10, 1962, p. 53.
2. N. C. Miller and G. A. Shirn, "Co-sputtered Cermet Films," *SCP and Solid State Technol.* **10**, 28 (1967).
3. M. Beckerman and R. E. Thun, "The Electrical and Structural Properties of Dielectric Metal Mixtures," *1961 Transactions of the Eighth National Vacuum Symposium* (Washington, DC) **2**, 905 (1962).

4. R. Wagner and K. M. Merz, "Stable, High Range Cr/SiO Film Resistors," *Proceedings of the Electronic Components Conference*, Washington, DC, May 5-7, 1964, p. 97.
5. R. Glang, R. A. Holmwood, and L. I. Maissel, "Sheet Resistance Control of Thin Cr-SiO Films," *Thin Solid Films* **1**, 151 (1967).
6. J. S. Logan, "Control of RF Sputtered Film Properties Through Substrate Tuning," *IBM J. Res. Develop.* **14**, 172 (1970).
7. The buffered hydrofluoric acid solution was obtained from the Transene Co., Inc., Rowley, MA.
8. L. I. Maissel and P. M. Schaible, "Thin Films Deposited by Bias Sputtering," *J. Appl. Phys.* **36**, 237 (1965).
9. J. D. Mackdougall, K. E. Manchester, and P. E. Roughan, "High Value Implanted Resistors for Microcircuits," *Proceedings of the IEEE* **57**, 1538 (1969).
10. R. Glang, R. A. Holmwood, and S. R. Herd, "Resistivity and Structure of Cr-SiO Cermet Films," *J. Vac. Sci. Technol.* **4**, 163 (1967).

Received April 7, 1977; revised June 20, 1977

T. Ushiroda is located at the IBM Fujisawa development laboratory, 1, Kiri-hara-cho, Fujisawa-shi, Kanagawa-ken 252, Japan. H. Matino is on leave from the above location and is presently at the IBM Thomas J. Watson Research Center, Yorktown Heights, New York 10598.

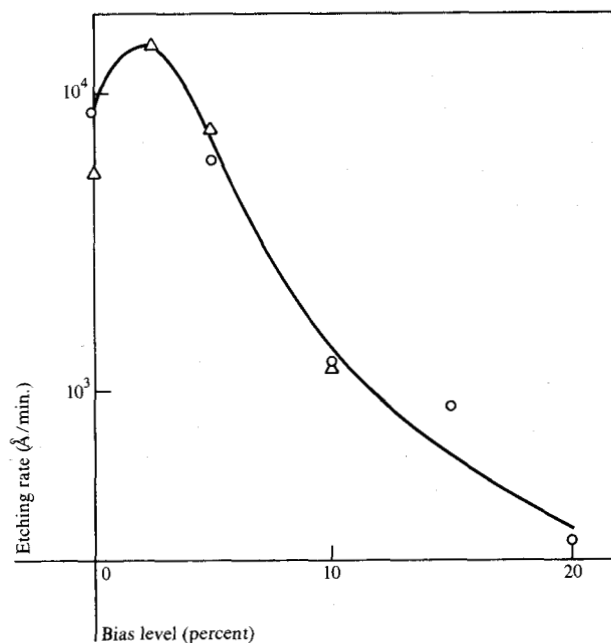


Figure 4 Etching rate (at 293 K) as a function of the percent of bias voltage for 0.5 kW (Δ) and 1.0 kW ($\circ$) rf power levels.